Magnetic Measurements with Metal Film Cantilevers: A User's Guide

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Abstract

High sensitivity magnetometry with cantilevers is possible at high magnetic fields, but for the uninitiated results of such measurements can be confusing or misleading. Many users who visit the National High Magnetic Field Laboratory experience for the first time the challenges of interpreting the results of measurements with cantilevers. What follows is a basic introduction to magnetometry with metal film cantilevers. A number of problems associated with this technique will be examined, including: (1) selection of appropriate cantilever for the application, (2) avoiding common methodological errors in data taking, and (3) interpretation of data. Examples of actual measurements will be discussed.

1 Magnetometry at the NHMFL

At the National High Magnetic Field Laboratory(NHMFL) a variety of standard instrumentation for carrying out magnetic measurements is available to users. Available magnetometers include: a Lakeshore vibrating sample magnetometer(VSM) modified for use in the resistive magnets, a selection of AC

Table 1: Comparison of the various magnetometers available at the NHMFL.

Inst.	Max. H (T)	T Range (K)	Sens. (EMU)
VSM	33	0.3 - 550	10^{-3}
Non-translating AC Susceptometer	33	0.02 - 300	10^{-4}
Translating AC Susceptometer	33	0.02 - 300	10^{-6}
Kerr Probe	27	2 - 300	10^{-9} - 10^{-10}
Si Cantilever	33	0.02 - 300	10^{-7} - 10^{-9}
Metal Cantilever	33	0.02 - 300	10^{-3} - 10^{-8}
Quantum Design SQUID	6	0.7 - 300	10^{-6}

susceptibility coils for a wide range of sample sizes, a probe for measuring the magneto-optical Kerr effect, and an assortment of cantilever magnetometers. A summary of the available magnetometers is found in Table 1. In addition to the standard instrumentation, the NHMFL staff will work closely with a user to develop new instrumentation to address particular experimental problems which demand extrinsic conditions or sensitivities not covered in the standard instrumentation.

The following paper serves as an introduction to making magnetic measurements with metal film cantilevers. This guide will begin with a general introduction to cantilever magnetometry and then go on to discuss the details of making the measurement, collecting the data, and then analyzing the results.

2 Cantilever Magnetometry

Cantilevers are used for a variety of different measurements: magnetic imaging, infrared radiation detection [2], measurement of fluid flow, acoustic detection, magnetometry, etc. This paper focuses on the application of cantilever technology to magnetic measurements.

The general principle behind all cantilever magnetometry is that a magnetic sample mounted on a flexible beam will respond to the application of a quasi-static magnetic field by experiencing a force and a torque which deflects the flexible beam. This technique was developed by Brooks *et al.*at

MIT in the 1980s [1]. The magnitude of the force and torque is related to the magnetization of the sample.

$$F = M \cdot \frac{dB}{dz} \quad (1)$$

$$\tau = M \times B \quad (2)$$

The deflection of the flexible beam can be measured in a number of ways: (1) capacitively[3], (2) piezo-resistively[4], (3) optically (e.g. via a diffraction grading), etc. If the magnetic moment of a sample is anisotropic, then measurements of the torque are more sensitive than force measurements. The force on a sample in a field gradient of 0.1 T/cm is typically smaller than the torque. Hence, cantilever magnetometry is often called torque magnetometry. The sensitivity and resolution of cantilever magnetometers depends on the stiffness of the flexible beam material, the sensitivity of the cantilever to temperature fluctuations and mechanical vibration, the quality of the measurement instrumentation, and the magnitude of the applied field. Typical sensitivities range from 10^{-9} to 10^{-11} N or 10^{-7} EMU (10^{-10} J/T measured in a 0.1 T/cm field gradient) to 10^{-9} EMU for a torque measurement at 20 tesla. The size of a cantilever varies depending on application from 50 to 100 μm long like those used for atom force microscopy to 5 mm long like those used at the NHMFL for magnetometry measurements on bulk samples. Shape also depends on application, but in general cantilevers exhibit a diving board-like structure; however, other related structures such as "teeter-totter" or "trampoline" are common for various applications.

In principle it is possible to extract calibrated measurements of magnetization from a cantilever magnetometer; however, in practice absolute measurements are difficult. Methods of calibrating cantilevers will be discussed below. Relative measurements of magnetization are easily done with cantilevers. Cantilevers are ideal for the measurement of quantum oscillatory phenomena and magnetic phase transitions such as critical points in superconductors, metamagnetic phase transitions, spin-Peierls transitions, etc. Examples of such measurements will be given below.

3 Design

This section focuses on the design and construction of a capacitive cantilever magnetometer. Many designs are possible. What follows is a description of

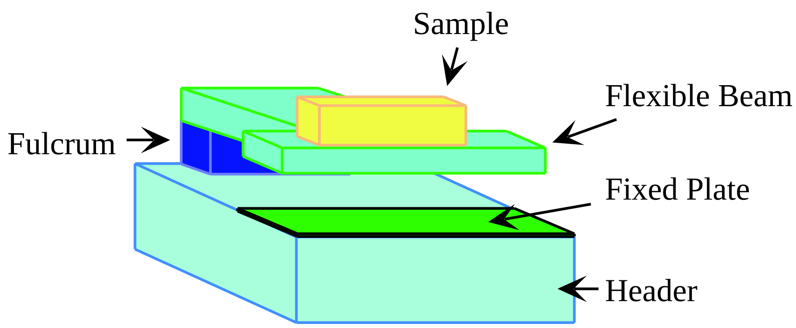

Figure 1: Shown here are the basic parts of a cantilever magnetometer. The sample is attached to the flexible plate (typically with a small amount of Apiezon N grease). The thickness of the flexible plate varies depending on the magnetization of the sample. The fixed plate is made out of metal and is permanently attached to the header. Headers at the NHMFL are machined from G10 fiberglass. The fulcrum can either be a part of the flexible plate (as it is in the Tau Sensors silicon beam cantilever) or part of the header.

the instrument used at the NHMFL.

3.1 Parts and Structure

A cantilever magnetometer consists of five parts: (1) the flexible beam, (2) the fixed plate, (3) the header, (4) the sensor mounting stage, and (5) the grounding can. A simple drawing of a cantilever magnetometer is shown in Fig. 1. Materials for the flexible beam are discussed in section 3.2. The header attaches to the sensor mounting stage. The purpose of the mounting stage is to provide a platform for placing thermometers and heaters near the sample. A hall probe or astatic pair can also be incorporated into the sensor mounting stage to assist in the determination of field center. The entire cantilever magnetometer is encased in a grounding can that serves as part of the third terminal in the three terminal capacitance measurement.

3.2 Flexible Materials

Many different materials can be used to construct cantilever magnetometers. Early measurements were made by suspending metallic samples on a thin gold or copper wire. Thin glass slides with a layer of gold deposited on

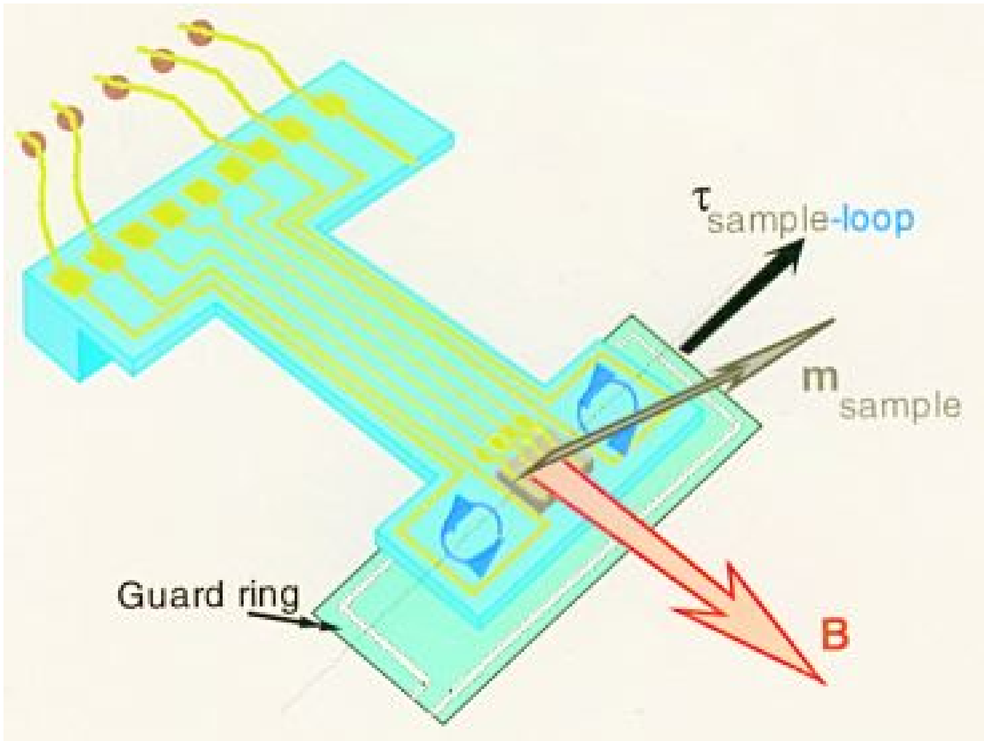

Figure 2: This cantilever is fabricated from silicon. Compensation loops and transport measurement wires are provided. Such cantilevers are available from Tau Sensors Ltd. or from Oxford Instruments. This technology was developed by Michael Naughton. The Tau Sensors cantilever is available to users of the NHMFL DC field facility.

one side have also been used. Today commercial cantilevers are fabricated from silicon wafers and are patterned with compensation loops and wires for transport measurements 2. While silicon cantilevers are highly sensitive, they are also highly expensive and fragile. Because silicon cantilevers are easily broken, it is useful to make initial measurements on a sample whose magnetic properties are unknown with a metal film cantilever. Metal film cantilevers are cheap (pennies per device as opposed to thousands of dollars per device for silicon cantilevers) and almost indestructible. This is not to say that metal film cantilevers replace their silicon and GaAs counterparts; metal films are complementary to the silicon devices in a user facility. *Unless the magnetic properties of a sample are well known before the beginning of*

Table 2: A summary of the materials and thicknesses presently available at the NHMFL for metal film cantilevers.

Material	Thickness (in.)	Note
BeCu	0.00055	non-magnetic
MP35N	0.0003	not heat treated
MP35N	0.0003	heat treated (stiffer)
Phosphor Bronze	0.002	
Phosphor Bronze	0.003	

a measurement, a metal film cantilever should be used prior to making the measurement with a silicon cantilever.

Development of metal film cantilevers at the NHMFL was driven by more than just economic factors. A real sensitivity gap existed in the magnetometry instrumentation available at our DC field facility. With a VSM adapted for use in the Bitter magnets, sensitivity of less than 10^{-3} EMU is difficult. With the silicon cantilever, magnetic moments larger than 10^{-5} EMU could damage the instrument. Hence, cantilever fabricated from very thin metal films helped closed this sensitivity gap. Additionally, measurements on superconducting samples with large diamagnetic signals could be carried out.

At the NHMFL a number of metal films are available for use as cantilevers; the materials are summarized in Table 2.

The stiffness of thicker metal film cantilevers allows the experimenter to make use of larger samples. Thinner cantilevers can be bent into the non-Hooke's law response region by the pull gravity on a massive sample.

4 Measurement Methods

Samples can be attached to the metal film cantilever with a spot of Apiezon N grease. According to a report in the summer 1999 issue of the trade magazine *C*oldFacts, Apiezon N has a better thermal conductivity at low temperatures than other grease. The sample should be mounted close to the tip of the cantilever, as far from the fulcrum as possible. Centering of the sample on the cantilever is desirable.

As mentioned in the introduction, when a magnetic field is applied to the sample attached to the flexible plate, it experiences both torque and force.

If the homogeneity of the magnet is good, the force term can be minimized by placing the sample in field center. This will result in a signal that is proportional to the torque on the sample. If the sample is isotropic or you wish to examine the component of magnetization parallel to the applied field, it will be necessary to carry out measurements in a field gradient. In this case the force measurements should be carried out a two different positions so that contributions from the torque can be minimized (at least to a first approximation). The experimenter should be aware that even in magnetically isotropic materials, the shape of a sample will introduce enough anisotropy that significant torques will be produced.

Metal film cantilevers do not have compensation loops. In some cases compensation loops may improve a measurement, but the use of compensation loops in the silicon cantilevers also has limitations. A current generated in the compensation loop will create a magnetic dipole. The strength of this dipole can be tuned so that its magnitude will equal that of the magnetic moment of the sample. The dipole generated in the compensation loops can be tuned by a feedback circuit so as to hold the capacitance of the cantilever constant. This method has a limited dynamic range, it leads to heating of the sample from the current running in the loops, and the background noise increases due to pickup in the loops of the field ripple and unwanted induced currents from vibrations. The main benefit to using compensation loops is to keep the sample at a fixed position with respect to the applied field. This can get rid of torque interaction in de Haas-van Alphen measurements or make it possible to maintain the cantilever response in the Hooke's law region. However, it does not remove the effects of torque and force component mixing.

Measurement of the capacitance of the cantilever magnetometer is made with a 1616 General Radio capacitance bridge. Any lock-in amplifier can be used to drive the bridge and detect the imbalance signal. Typical drive frequencies range from 1 kHz to 10 kHz (for work in DC fields). Bridge excitation voltages range from 1 to 30 V_{rms}. The lock-in time constant can be short (10 ms and 12 dB/oct) for quick response, but longer integration times are helpful in some cases where signals are small. Typical measured capacitances for the metal film cantilevers at the NHMFL are 0.9 to 1.1 pF.

A rule of thumb when making measurements is to monitor the total capacitance change as the field is ramped up. Changes of more than 10 percent of the zero field capacitance.

Because the MP35N contains Ni, it is magnetic. How much this affects a measurement depends, of course, on the size of magnetization of the sample place on the cantilever. Figure 3 shows the field dependence of the MP35N cantilever.

All cantilevers are temperature dependent. Fig 4 shows the temperature dependence of the MP35N cantilever.

5 Data Analysis

This section outlines suggested approaches for analyzing data taken with the metal film cantilever.

5.1 Magnetization Curves

Extracting the dependence of magnetization as a function of field from cantilever data requires attention to detail. Background measurements of a bare cantilever should be done at the fields and temperatures at which the sample will be measured. These background traces must be subtracted from the data. Figure 5 shows a comparison of data taken on $LiCu_2O_2$with the VSM and with a metal film cantilever. As can be seen in the figure, the metal film cantilever data, although it has been corrected with the background trace, still differs from the VSM data because the torque signal has not been subtracted from the data. To properly correct this magnetization versus field data, a background trace with no sample, and measurements at two different gradients, must be taken. Note, that applied field values also need correcting when measuring in a field gradient.

5.2 Phase Transitions

Metal film cantilevers are ideal for measurement of phase transitions that are too small for detection with AC susceptibility. For example, measurements of the phase boundary $T_q(H)$between the anti-ferro-quadrupolar state (AFQ) and paramagnetic state in CeB_6have been carried out with a metal film cantilever. An example of the raw data is shown in Figure 6. Plotting $T_q$as a function of applied field yields the phase boundary shown in Figure 7.

The primary difficulty with the measurement of phase transitions with cantilevers is temperature measurement and control. Because cantilevers are highly sensitive to temperature change, in fields higher than 19 tesla,

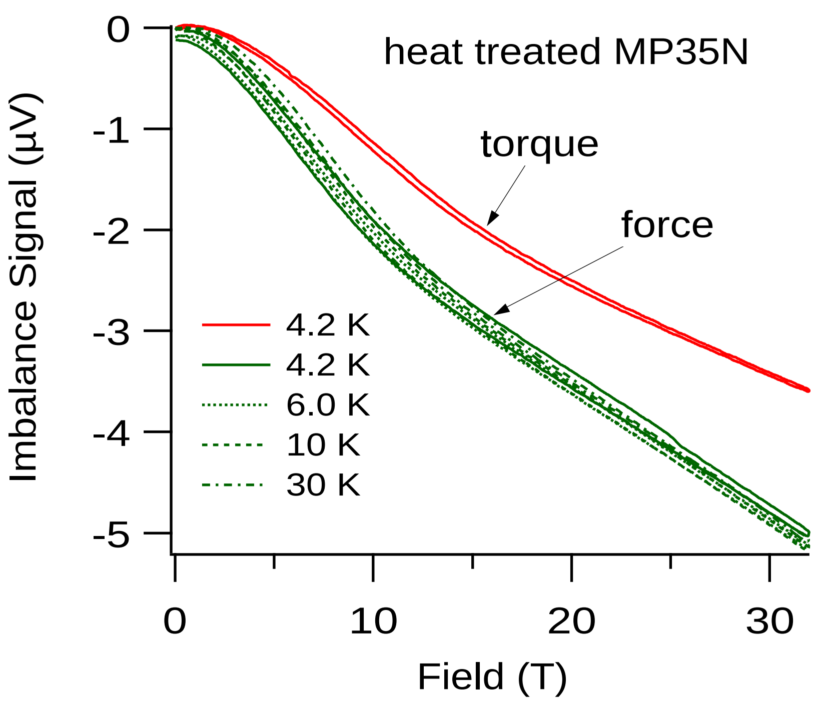

Figure 3: A bare heat treated MP35N film cantilever was measured as a function of field at several temperatures. Torque and force measurements yield a similar background trace, differing only in magnitude of signal. From the measurements in the field gradient at different temperatures, it is clear that this cantilever's field dependence does not change significantly as a function of temperature. The imbalance signal was measured with a 5 kHz, 10 V_{rms} excitation of the capacitance bridge.

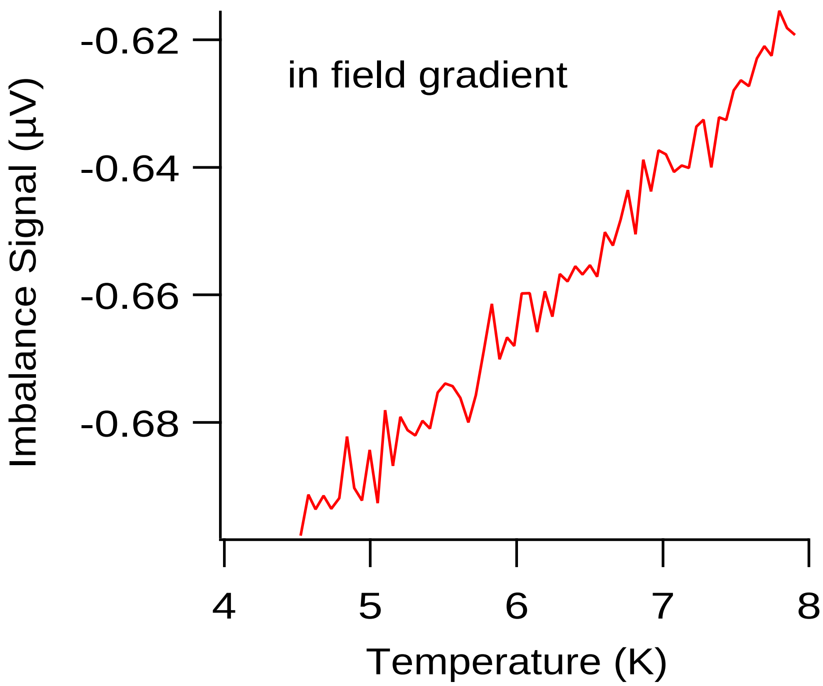

Figure 4: A bare heat treated MP35N film cantilever was measured in a field gradient (with the main field set at 4 tesla) as a function of temperature. The temperature dependence of these cantilevers is approximately linear at low temperatures.

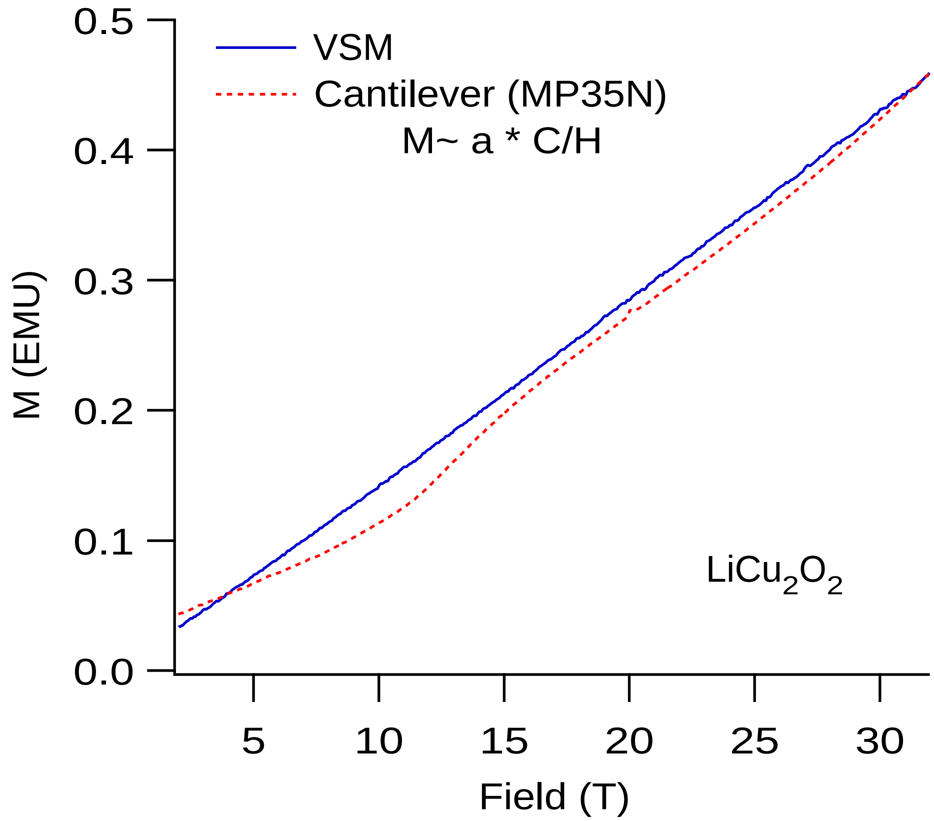

Figure 5: Magnetic measurements on $LiCu_2O_2$made with a VSM and a metal film cantilever. The cantilever data has been corrected. The background was subtracted and the capacitance signal was divided by the applied field, then scaled to the VSM data at 32 tesla. The deviation of the cantilever data from the VSM curve is due to the uncompensated torque signal.

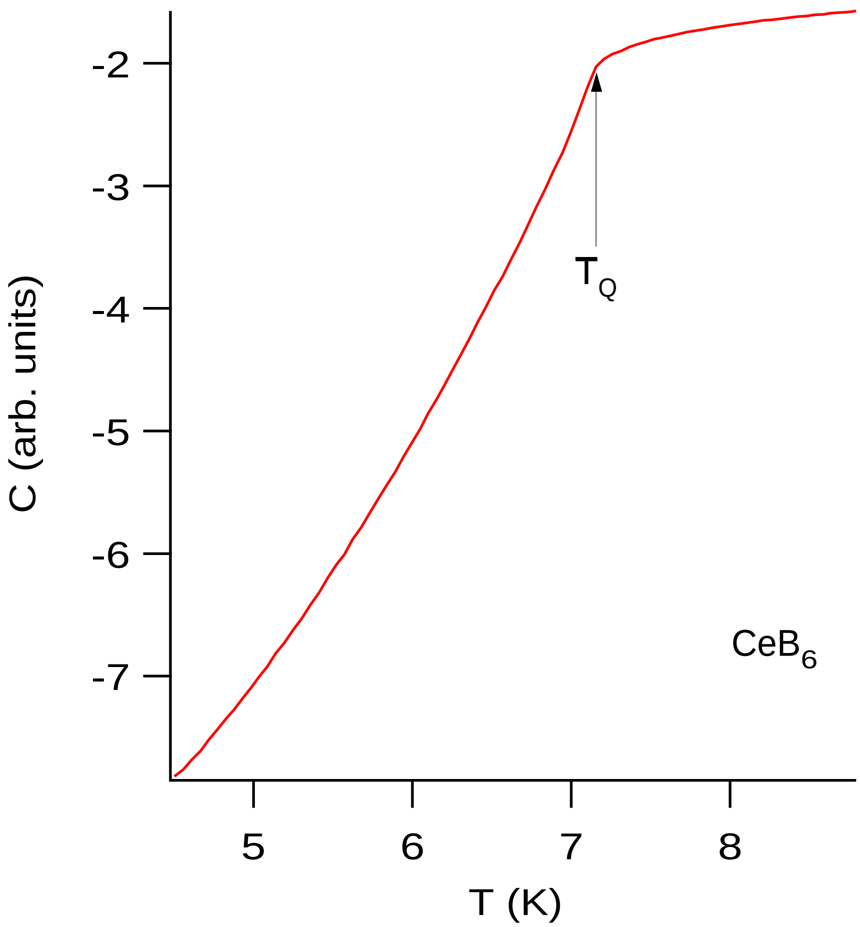

Figure 6: Capacitance is shown as a function of temperature for a sample of CeB_6measured with a heat treated MP35N cantilever. The bend in the curve represents the phase transition $T_q$between the AFQ state at low temperatures and the high temperature paramagnetic state.

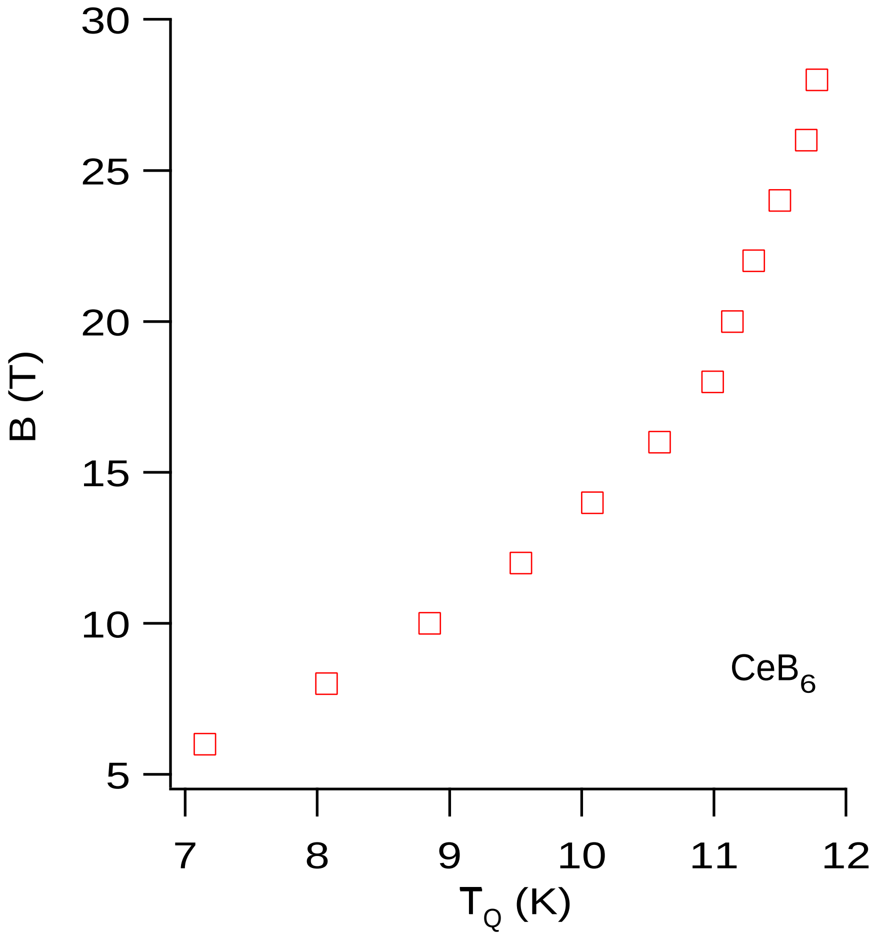

Figure 7: T_q(H)is plotted as a function of the applied field.

measurements are complicated by the trapping of helium bubbles at the maximum gradient positions. Measurements carried out in helium gas will likely be complicated by a temperature gradient between the sample and the thermometer unless great care is taken to thermally sink the thermometer to the cantilever flexible plate.

5.3 Hysteresis Measurements

Metal film cantilevers have also been used successfully to measure hysteresis in superconducting samples. From the hysteresis curves critical points can be extracted, but the thermodynamic critical field cannot be extracted since integration under the hysteresis curve is not meaningful.

Measurements of the hysteresis in $Ba_{1-x}K_xBiO_3$are shown in Figure 8. The reversibility field $H_r$is shown in Figure 9. Data for the reversibility field as a function of temperature is shown in Figure 10.

5.4 de Haas-van Alphen Effect

Finally, metal film cantilevers are excellent for the measurement of de Haas-van Alphen(dHvA) oscillations. An example of dHvA oscillations is shown in Figure 11.

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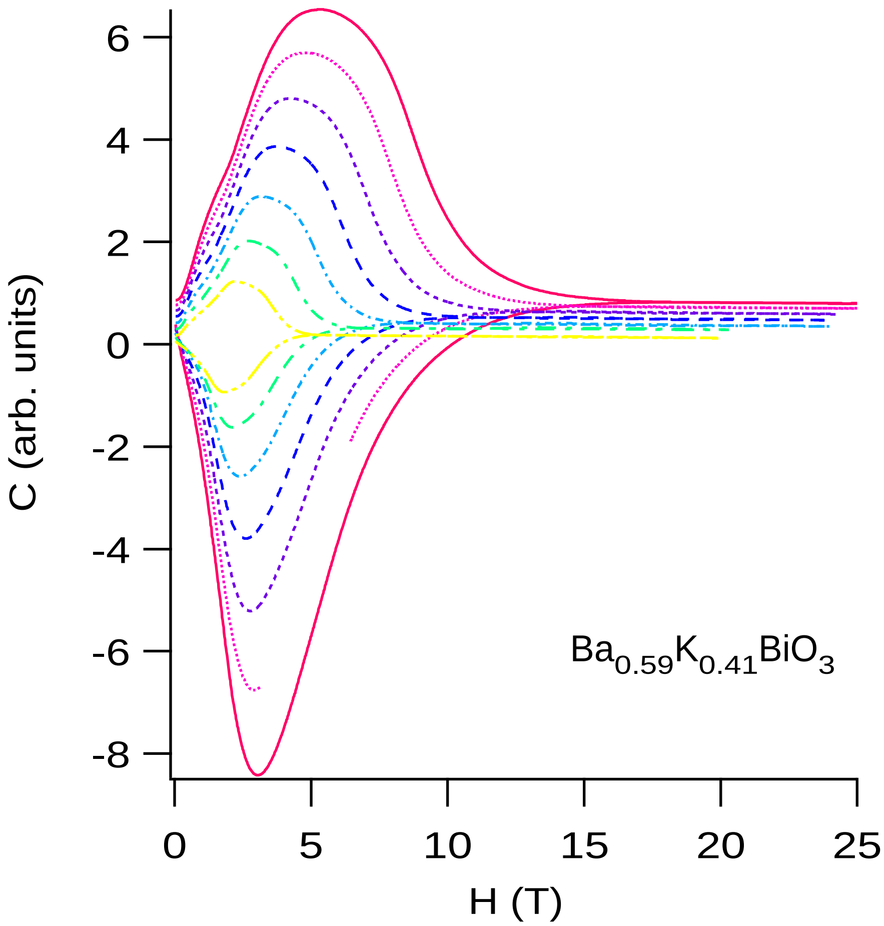

Figure 8: Several hysteresis curves for a sample of BKBO are shown as a function of field. The different curves are measurements at different temperatures.

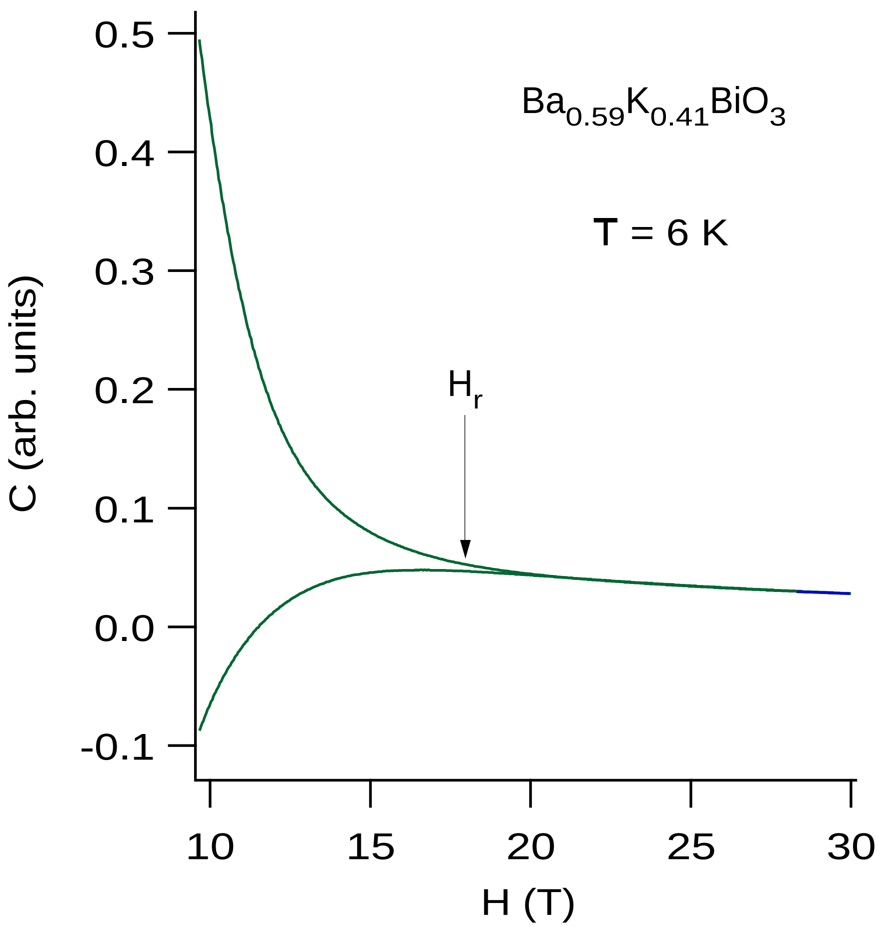

Figure 9: H_r is defined as the field at which the down field sweep deviates from the up field sweep.

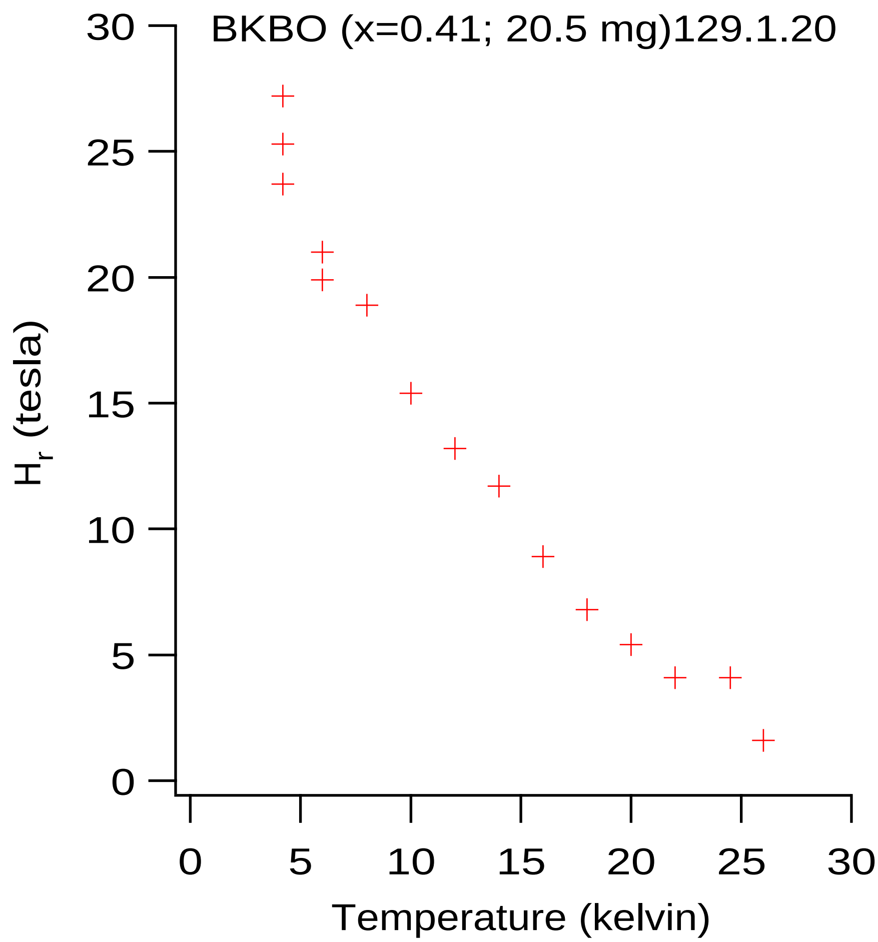

Figure 10: $H_r$is shown as a function of temperature.

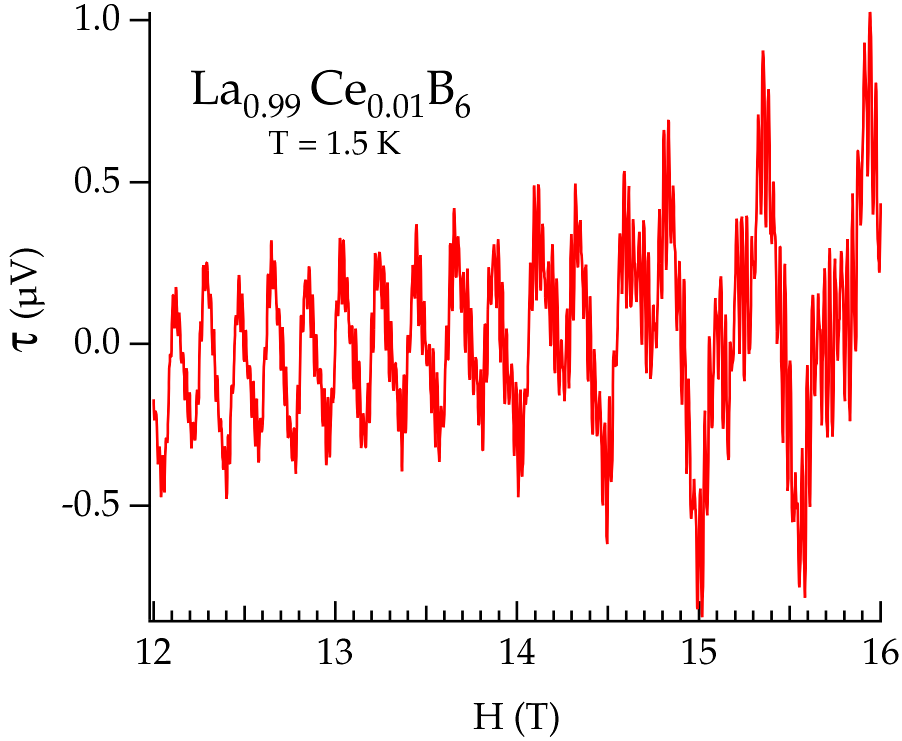

Figure 11: dHvA oscillations in Ce doped LaB_6. The measurements were carried out at field center and at 1.5 K. The torque is shown in terms of the imbalance signal from the capacitance bridge.